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METHOD OF LEAST SQUARES APPLIED TO ESTIMATING ERRORS IN COAL ANALYSIS

BY

J. D. DAVIS AND J. G. FAIRCHILD

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METHOD OF LEAST SQUARES APPLIED TO ESTIMATING ERRORS IN COAL ANALYSIS.

By J. D. Davis and J. G. Fairchild.

INTRODUCTION.

In this paper, which is published by the Bureau of Mines as a contribution to the increase of efficiency in the utilization of fuel, the authors have sought to describe a definite method for estimating the probable limits of error in analyzing samples of coal, and to show how this method, that of least squares, should be applied.

Bailey [a] has shown that the method of least squares is applicable to the problem of sampling coal; it can be used to determine the limits of accuracy of sampling and for preparing standardized methods of sampling, but so far as the authors know the accuracy of the ordinary methods of analyzing coal has not yet been investigated by a mathematical method. It is customary to estimate the limits of accuracy of analysis by observing the agreement of duplicate determinations; that is, from general experience with a given kind of analysis. Hence, as some sets of duplicates are found to agree more closely than others, this question arises: What is the widest variation and what is about the average variation reasonably to be expected? Or, in other words the question is: What is the magnitude of the "probable error" of a single determination and what is the maximum error to be expected, say, once in 1,000 determinations?

Although by the method of least squares direct expressions may be obtained for the magnitude of the errors in the work studied, the authors do not wish to imply that ideas formed from long experience are of no value; the theory of probability and the method of least squares are based on just such experience. Leplace has said "the theory of probability is nothing but common sense reduced to calculation." The chief value of the theory is that it furnishes a ready means by which the individual may obtain for himself a concrete expression of the precision of his own work and at the same time a convenient form of statement for the benefit of others.

The application of the theory of probability to obtain expressions for the accuracy of the proximate analysis of coal is complicated

[a] Bailey, E. G. Accuracy in sampling coal: Jour. Ind. and Eng. Chem., vol. 1, March, 1909, p. 161.

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through the fact that the same methods are used for all kinds of coal and for no two coals will the several constituents, as stated, represent exactly the same quantities chemically. Consequently the error is often a function of the stated chemical composition of the constituent. For example, if the mineral matter of a coal contains carbonates in varying percentages the probable error of the ash determination will be greater if the percentages of the carbonates are not determined and the proper correction made.[a]

Further, it is obvious that the analytical error will be to some degree a function of the quantity of the constituent determined. A rigid treatment would involve the examination of all classes of coals, and of coals from every bed represented, and would require an enormous amount of analytical work.

The authors have attacked the problem by first carefully working out the limits for a few coals that are selected as typical and then comparing the results so obtained with data compiled from numerous check determinations taken consecutively from the laboratory records. The number of check determinations was usually 1,000. The experimental work was all done by the same analyst; hence the effect of the personal equation was eliminated from the results. On the other hand the data taken from the records include the work of several analysts, so that the personal equation was here involved. Further, the experimental work for a given result took only a day or two, whereas the data represented in the compilation from the records represented at least a month's analyses, so that variations chargeable to changing conditions in the laboratory probably were involved in the recorded results used.

The committee on coal analysis of the American Society for Testing Materials has designated [b] limits of accuracy for each determination involved in coal analysis, and as this information represents the consensus of opinion of leading American chemists, the limits are given to serve as a standard of reference for the work that follows. The accuracy of the method of preparing laboratory samples should be checked frequently by resampling the rejected parts of the large (3 to 5 pounds) sample and preparing duplicate laboratory samples. Results for ash in duplicate samples should agree within the following limits:

Limits of error of ash determinations for duplicate samples.

	Per cent.
No carbonates present	0.4
Considerable carbonates and pyrite present	.7
More than 12 per cent ash and containing considerable carbonate and pyrite	1.0

[a] Parr, S. W., Preliminary report of the committee on coal analysis: Jour. Ind. and Eng. Chem., vol. 5, June, 1913, p. 523.

[b] American Society for Testing Materials, Yearbook for 1915, p. 596.

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The "allowable variations" in analysis of small laboratory samples are as follows:

"Allowable variations" in coal analysis, as designated by American Society for Testing Materials.

	Same analyst, per cent.	Different analysts, per cent.
Moisture determination.		
Moisture under 5 per cent	0.2	0.3
Moisture over 5 per cent	0.3	0.5
Volatile matter determination.		
Bituminous coals	0.5	1.0
Lignites	1.0	2.0
Ash determination.		
No carbonates present	0.2	0.3
Carbonates present	0.3	0.5
More than 12 per cent ash and containing carbonates and pyrite	0.5	1.0
Sulphur determination.		
Sulphur under 2 per cent	0.05	0.10
Sulphur over 2 per cent	0.10	0.20
Calorimeter determination.		
All coals	0.3	0.5

The term "allowable variations" should be construed not strictly in the sense that agreement within the prescribed limits should always be obtained with ordinary care in manipulation, but rather that the great majority of check determinations will agree within the limits given; the limit itself should be taken merely as a criterion of the desirability of repeating the determination.

PREPARATION OF THE SAMPLE OF COAL FOR ANALYSIS.

Samples of coal for analysis are received by the Bureau of Mines in one or more galvanized-iron cans containing when full about 3 pounds each. The method of reducing a sample to a size and bulk convenient for making the analysis is as follows:[a]

The entire sample, the coarsest particles of which will pass a 4-mesh screen ($\frac{3}{16}$-inch holes), is first crushed by a roll crusher, a grab sample for the total moisture determination is taken, and the whole is put through a 20-mesh sieve. Any particles too coarse to pass the sieve are again run through the crusher. The bulk of this 20-mesh sample is now reduced to about 6 ounces by a Taylor & Brunton splitter (Pl. I). The 6-ounce sample is further crushed by a planetary disk crusher after which it is divided on the splitter. The resultant sample (about 3 ounces), which will all pass a 60-mesh sieve, is placed in a 4-ounce rubber-stoppered bottle for analysis.

[a] Stanton, F. M., and Fieldner, A. C., Methods of analyzing coal and coke: Tech. Paper 8, Bureau of Mines, 1913, p. 1; Pope, G. S., Sampling coal deliveries: Bull. 63, Bureau of Mines, 1913, p. 33.

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As the precision attainable by this method of reduction depends largely on the distribution of the impurities in the sample, the authors sought at the outset to gain some idea of the variation of impurities in different parts of the sample. The separation of impurities by the gravity or washing method was considered, but as that method seemed particularly adapted to the somewhat complete separation of extraneous ash (slate, pyrite, or bone), the variations indicated would hardly be approached in the method of reduction applied. It seemed reasonable to assume that some of the materials making up the sample would crush more easily than others; that the coal substance itself would perhaps crush more easily than the impurities. If the ash determination be used as a measure of the impurities present in different parts of a sample, the maximum variation attainable by sizing would then be evident, and would serve as an indication of the difficulty of obtaining a correctly representative sample. It was recognized that the distribution of impurities might well be affected by factors other than the resistance to crushing and that even the crushing factor for the same kind of impurities would probably vary with the condition of the coal, for instance with the moisture content. Accordingly it was not expected that a law could be found connecting the size of particles with the ash content. The results of tests tabulated in Table 1 and shown in part graphically by figure 1 would seem to be in accord with this reasoning.

Six samples of coal selected at random were crushed to 20-mesh and screened as shown in Table 1, following. Each screened part was weighed and analyzed (duplicate determinations) for moisture and ash, the "weighted mean" of all determinations being taken as the true value for the original gross sample. The "weighted mean" was obtained by multiplying the determined percentage of moisture or ash by the weight in grams of the sample represented, adding the results, and dividing the sum by the total weight of the gross sample. The sixth and seventh columns of the table show the variations from the true value for each sized part of the sample.

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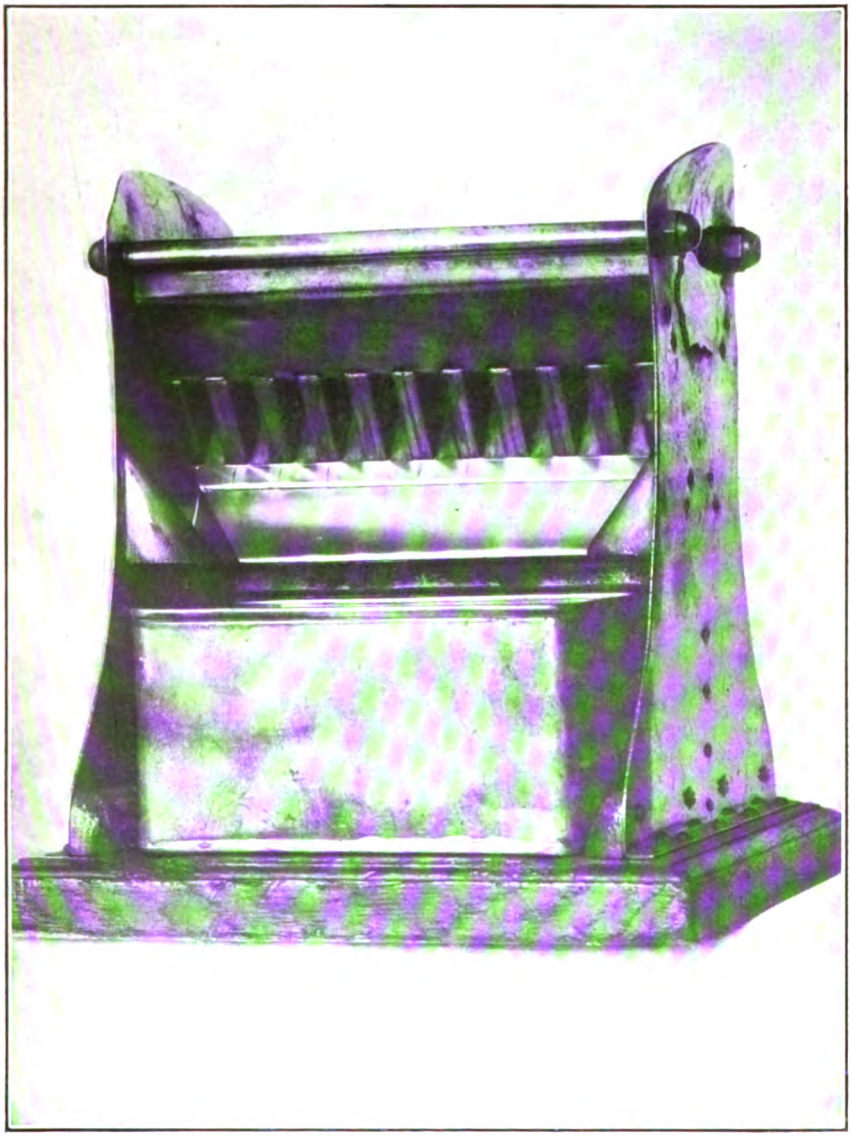

TAYLOR & BRUNTON SPLITTER FOR REDUCING COAL SAMPLES.

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TABLE 1.—*Results of analysis of samples of coal from different stages of sizing.*

BITUMINOUS COAL FROM DUNSMUIR, VANCOUVER ISLAND, B. C. (SAMPLE 57831).

Mesh.	Weight.	Moisture.	Ash in coal as received.	Ash in dry coal.	Moisture difference.	Ash difference.
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
On 40	272	2.22	11.98	12.25	+0.13	+0.27
On 60	152	2.10	10.92	11.15	+ .01	− .83
On 80	142	2.07	10.87	11.10	− .02	− .88
On 100	38	1.82	10.82	11.02	− .27	− .86
Through 100	268	2.00	12.52	12.78	− .09	+ .80
Mean		2.09		11.98		

ANTHRACITE BUCKWHEAT (SAMPLE 58077).

On 40	241	5.30	17.50	18.48	+0.27	−2.16
On 60	106	5.16	17.49	18.45	+ .07	−2.19
On 80	95	4.81	18.76	19.72	− .28	− .92
On 100	35	4.64	19.83	20.80	− .45	+ .16
Through 100	221	5.01	23.17	24.40	− .08	+3.76
Mean		5.09		20.64		

ANTHRACITE PEA (SAMPLE 58051).

On 20	4	2.42	12.39	12.70	−0.20	−2.67
On 40	584	2.84	14.28	14.63	+ .22	− .74
On 60	152	2.73	14.09	14.49	+ .11	− .88
On 80	102	2.62	14.48	14.88	.00	− .49
On 100	40	2.28	15.82	16.18	− .34	+ .81
Through 100	288	2.17	17.06	17.44	− .45	+2.07
Mean		2.62		15.37		

SEMIBITUMINOUS (SAMPLE 58131).

On 40	288	2.05	6.20	6.33	+0.37	+0.55
On 60	136	1.65	5.80	5.89	− .03	+ .11
On 80	104	1.36	5.32	5.39	− .32	− .39
On 100	40	1.07	5.28	5.34	− .61	− .44
Through 100	252	1.50	5.22	5.31	− .18	− .47
Mean		1.68		5.78		

LIGNITE (SAMPLE 57588).

On 20	4	35.64	6.03	9.37	−1.03	−1.36
On 40	428	40.23	6.04	10.10	+3.56	− .63
On 60	108	37.05	7.28	11.56	+ .38	+ .83
On 80	60	36.82	6.98	11.05	+ .15	+ .32
On 100	44	36.20	7.35	11.52	− .47	+ .79
Through 100	116	36.65	7.65	11.88	− .02	+1.15
Mean		36.67		10.73		

PEERLESS NO. 1 POCOHONTAS (SAMPLE 57454).

On 40	184	0.88	5.61	5.66	+0.18	+0.38
On 60	108	.73	5.00	5.04	+ .03	− .24
On 80	84	.63	4.73	4.76	− .07	− .52
On 100	48	.73	4.87	4.91	+ .03	− .37
Through 100	252	.58	5.32	5.35	− .12	+ .07
Mean		.70		5.28		

Figure 1 shows these results graphically. The ordinates give for each sample, in percentages, the positive and negative variations from the weighted mean, which is represented by the horizontal base line, and the abscissas show the mesh or fineness of the corresponding part of sample.

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As regards the anthracite coals, the fines invariably contained the most ash and the coarser sizes the least, the ash content of the intermediate sizes being near the mean value. For the bituminous coals

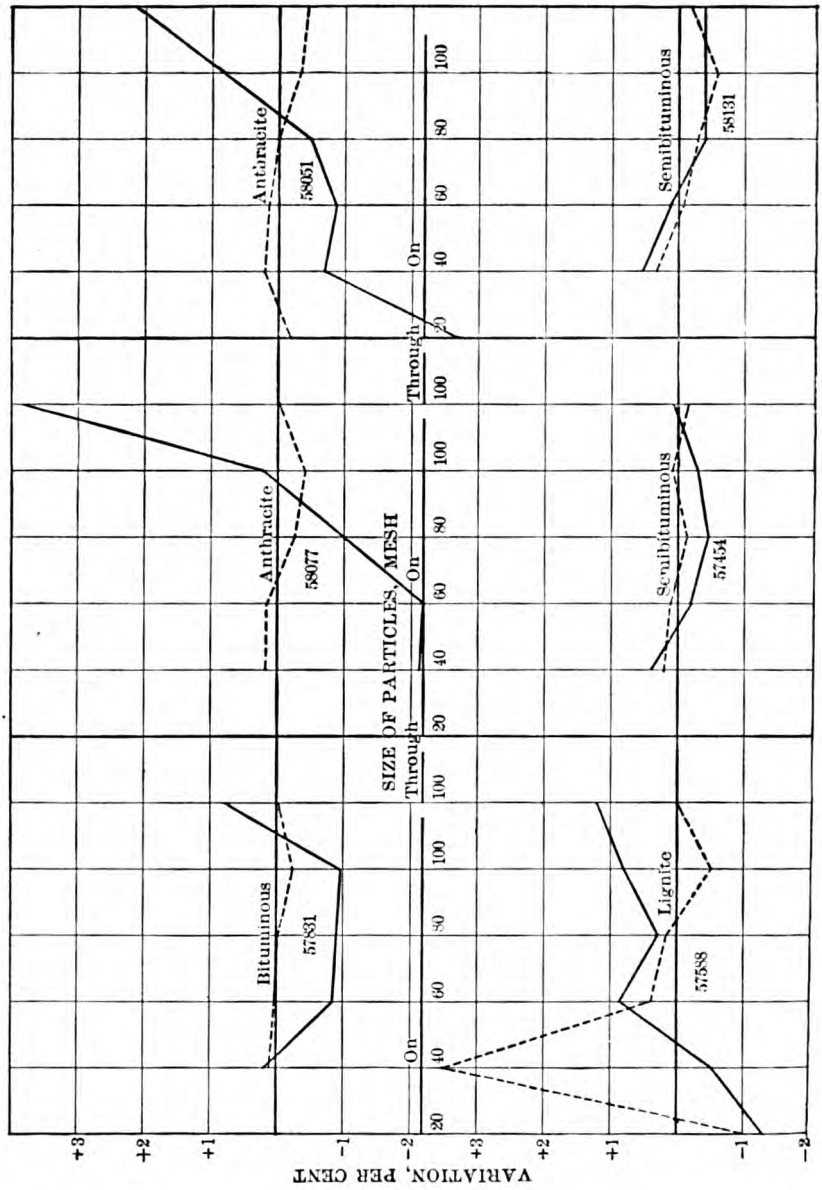

FIGURE 1.—Graphs of results given in Table 1. Moisture - - - -, ash ——.

there is no such regularity. Doubtless more data would bring out interesting points of regularity for various kinds of coal, but for the purpose of this paper the large amount of experimental work necessary

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hardly seems warranted, particularly as such data would apply only to crushing with rolls. In general, the order of variation that may be expected between different sizes is shown, and it is noteworthy that the differences are so large, particularly as concerns the coarse and fine material of the anthracite coals. In every sample the intermediate sizes were of lightest weight.

In this connection it was thought of interest to determine the eveness of distribution of the impurities in coal of a given fineness. Accordingly two coals were sized, 3-gram samples were taken at random from each sized part, and the ash content of each was determined. The results, which are given in Table 2 following, indicate that different parts of the same mesh sample vary considerably in ash content.

TABLE 2.—*Results of tests to determine uniformity of ash content in coal samples of a given fineness.*

BITUMINOUS COAL FROM MARION COUNTY, ILL. (SAMPLE 58211).

Mesh.	Ash content of grab sample No.—		
	1	2	3
	Per cent.	*Per cent.*	*Per cent.*
On 40	9.57	10.78	9.83
On 60	9.25	9.71	8.75
On 80	9.60	9.98	10.65
On 100	9.65	10.25	10.10

ANTHRACITE BUCKWHEAT (SAMPLE 68136).

On 40	21.62	21.83	22.38
On 60	23.45	23.55	22.00
On 80	23.88	24.68	24.05
On 100	24.32	24.65	24.65

For the particular bituminous coal selected the sizing seems to have little effect on the ash distribution, whereas with the anthracite coal its effect is evident. For the same mesh sample there is, however, considerable variation with both coals.

The importance of maintaining the proper ratio of weight of coarsest particles to total weight of sample has been demonstrated by Bailey,[a] who finds by experiment that the relation between "size-weight per cent," the probable error, and percentage of extraneous ash can be closely approximated by the parabolic equation:

$$r = .522\sqrt{ap} - .353p,$$

where r represents the probable error; that is, for a number of repeated trials there will be as many errors smaller than r as there will be greater; a represents the percentage of extraneous ash, or that coming from impurities in the coal, such as slate and pyrite; p represents the size-weight percentage, given by the ratio of the weight of the largest piece of slate or bone coal contained in the sample to the total weight of the sample.

[a] Bailey, E. G., Accuracy in sampling coal: Jour. Ind. and Eng. Chem., vol. 1, March, 1909, p. 171.

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This formula serves as a means of determining approximately the accuracy with which a sample has been prepared and also as a guide in estimating the proper amount of sample to take in any given case. Applying the formula to sample 58077 (Table 1), which shows the greatest variation of the series due to sizing, the error to be expected by the usual method of preparation of the sample may be found as follows: (1) Weight of largest piece of slate (20-mesh) equals 0.0036 gram; weight of smallest sample divided, equals 200 grams; size-weight per cent, 0.002, equals p; (2) assume a equals 6 per cent.

If these values be substituted in the formula, $r = 0.522\sqrt{ap} - 0.353p$, then r equals 0.056 per cent. This means that by the usual method of preparation, an error not greater than 0.056 per cent may reasonably be expected, whereas, if the sample had merely been screened with rejection of the coarser parts, an error of 3.76 per cent (Table 1) might have resulted. The effect of sizing is therefore manifest and the importance of keeping the sample well mixed at all stages of crushing and reduction is evident.

The significance attached to the moisture-content results presented in Tables 1 and 2 is that for this method of treatment the moisture content varies considerably with the variation in the size of particles. One might have expected the fines to hold the most moisture,[a] as none of the samples treated had been freshly crushed. However, large variations in moisture content would be caused by successive screenings, and hence no conclusions can be drawn.

It now becomes necessary to discuss briefly the theory of the law of error in order that the computations which follow be readily understood. A full discussion of the subject will be found in Merriman's book on least squares.[b]

For any given series of observations experience has shown that the following statements hold; that they are axiomatic: (1) Small errors are more frequent than large ones. (2) Positive and negative errors are equally frequent. (3) Very large errors do not occur.

From these axioms it will be evident that the probability of any error happening in a given series is a function of the magnitude of the error itself; that is, denoting the error by x and its probability by y, the equation $y = fx$ expresses the relation between the two. The curve, figure 2, following shows the relation graphically.

Here y, the probability, is measured along the ordinates and x, the error, is measured along the abcissas, the curve being symmetrical with respect to the y axis. It will be seen that for every positive value for x there is an equal negative value; that when x is small y is large, and that when x is very large y approaches zero in accord

[a] Fieldner, A. C., Notes on sampling and analysis of coal: Tech. Paper 76, Bureau of Mines, 1914, p. 23.
[b] Merriman, Mansfield, A textbook on the method of least squares, 1901, p. 13.

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with axioms 1, 2, and 3, given above. $\int x$ of the above equation admits of determination and may be shown to be equal to

$$ke^{-h^2x^2} \tag{1}$$

h and k being constants; k is the value of y when x equals zero and h is the measure of precision, so-called. For any series of observations where h of the one is larger than h of the other (y being the same in both cases), it will be seen that x must be the smaller in the former case. This series is therefore the more precise of the two.

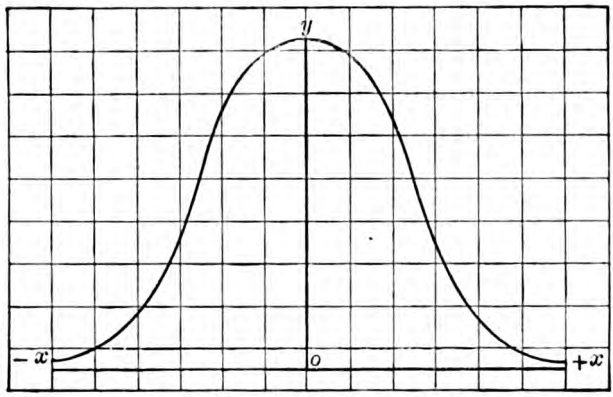

FIGURE 2.—The probability curve. $y = \int rx$.

If it is desired to know the probability of an error falling between limits $x = 0$ and $x = x'$, say, it may be found by integration of function (1) between limits 0 and x', thus:

$$P = \frac{2}{\sqrt{\pi}} \int_0^{hx} e^{-h^2x^2} dx h \tag{2}$$

the constant k being equal to $\frac{h}{\sqrt{\pi}}$. Values of this integral will be found tabulated in textbooks on the method of least squares[a] and will usually be found stated as

$$\frac{2}{\sqrt{\pi}} \int_0^t e^{-t^2} dt \text{ where } t = hx$$

Now the probable error, r, of a series of observations is defined as such that "it is an even wager that an error taken at random in the series will be greater or less than the probable error." Its probability is therefore one-half.

[a] Merriman, Mansfield, A textbook on the method of least squares, 1901, p. 220; Bartlett, Dana P., The method of least squares, 1915, appendix, p. 7; Smithsonian Physical Tables, 6th ed., 1914, p. 56.

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By solving the expression

$$\tfrac{1}{2} = \frac{2}{\sqrt{\pi}} \int_0^{hx} e^{-h^2x^2} dhx$$

and putting x equal to r, hr is found to be equal to 0.4769. (3)

The probability of the occurrence of independent errors x_1, x_2, x_3, or x_n is equal to the product of their separate probabilities, or (from equation 1)

$$P' = h^n (dx)^n \pi^{\frac{-n}{2}} e^{-h^2 \Sigma x^2}$$

from which, if P' is to be of maximum value, it is required that Σx^2 be small. Taking the first derivative $\frac{dP'}{dh}$ of the expression, placing the result equal to zero and reducing, it is found that, $n - 2h^2 \Sigma x^2 = 0$ or

$$h = \sqrt{\frac{n}{2\Sigma x^2}}; \text{ but from (3) } r = \frac{0.4769}{h}$$

whence

$$r = 0.6745 \sqrt{\frac{\Sigma x^2}{n}}$$

The value x, used heretofore, represents a true error, but the corresponding residual v which represents the difference between the arithmetical mean of a series of observations and a single observation may not be taken as the true error. The residual v may be said to differ from x by some small factor u, so that $\Sigma x^2 = \Sigma v^2 + u^2$ and v^2 may be taken as approximately equal to $\frac{\Sigma x^2}{n}$, from which $\frac{\Sigma x^2}{n} = \frac{\Sigma v^2}{n-1}$; r, the probable error, is then equal to

$$0.6745 \sqrt{\frac{\Sigma v^2}{n-1}} \tag{4}$$

which is the formula used in calculating r from the tables that follow.

Another value, u, is also used that may be called the high error for a given series. The probability of its occurrence is one in one thousand, and it is equal to 4.9 r, r being the probable error, found from expression 4.

To compute the probable error, r, of a series of observations it is only necessary to determine the arithmetical mean of the observations, find the differences v between individual observations and the mean, and then apply formula 4.

As regards the curves shown in figures 3, 4, and 5 (pp. 17, 29, and 32):

For the curves a, the values of r were those computed from the series of observations represented, the h values being found from the

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relation $hr = 0.4769$ (p. 14). The h values found were substituted in the equation,

$$y = \pi^{-\frac{1}{2}} h e^{-h^2 x^2}$$

from which for various assumed values of x the corresponding values for y were found and plotted. In order to facilitate plotting, the y values found were multiplied by the factors given on the margin of the sheets. Where such curves are plotted to the same scale, the height of the curves is a measure of their relative precision; that is, the higher the curve, the higher the degree of precision represented. All the curves shown are comparable except the highest curve in figure 5, page 32, which represents the highest degree of precision of the three curves and could not be plotted to the same scale as the other two. The ordinates shown for these curves equally divide the areas included between the curves and the axes, and the x values corresponding are the probable errors in each case.

Curves b show the relation between the magnitude of an error x, and the per cent of total number of errors for the series falling inside limit x. In plotting determined values for h and r are taken from the series represented, whence values of t in the integral

$$2\pi^{-\frac{1}{2}} \int_0^t e^{-t^2} dt$$

may be found for various assumed values of x. Values of P, the corresponding probability, may be read from tables of values of the integral. Values for P found in the tables multiplied by 100 give directly the per cent of total number of errors falling inside the limit x corresponding. The theoretical number of errors falling between limits given in Table 4 (p. 19) were calculated by the above method. Here the close agreement of the theoretical results with those obtained experimentally will be noted.

ERROR OF THE ASH DETERMINATION.

The method followed by the authors in making ash determinations on individual samples was that recommended by the committee on coal analysis of the American Society for Testing Materials,[a] which is as follows:

Place the porcelain capsules containing the dried sample from the moisture determination in a cold muffle furnace, or on the hearth at a low temperature, and gradually heat to redness at such a rate as to avoid mechanical loss from too rapid expulsion of volatile matter. Finish the ignition to constant weight (± 0.001 gram) at a temper-

[a] American Society for Testing Materials, Year Book, 1915, p. 665.

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ature between 700° and 750° C. Cool in a desiccator, and weigh as soon as cold.

As regards the determinations represented by the data taken from the records (fig. 3), the method mentioned was not followed strictly in that the ashing was sometimes started in a hot muffle; further, the temperature of the muffle frequently exceeded 750° C. Some of the later determinations, however, were made in a muffle provided with temperature-control apparatus with which the bureau's laboratory has recently been equipped. Results reported in Tables 3 and 4 were obtained by the method of ashing recommended by the committee. In this connection, and as a matter of interest, attention is called to the limit agreement (0.001), constant weight, as given. An analyst using this limit agreement may obtain an error as large as the total variation permitted between duplicates if a coal is low in ash and contains no carbonates. Further, as is presently shown, the limit agreement allowed is not less than the probable error of the ash determination as experimentally determined. If it be admitted that for certain coals it is difficult to obtain closer agreement, and that closer agreement is often unnecessary, it would seem advisable, for the sake of consistency, to extend the limit of allowable variation to correspond.

In order to obtain expressions for the accuracy of the ash determination for coals containing less or more than 12 per cent ash, two 100-gram 60-mesh samples that were known to contain ash as required were chosen, and 24 ash determinations were made for each. The arithmetical mean of the results was found in each case. The residuals, v, were then computed and the probable error found by the formula

$$r = .6745\sqrt{\frac{\Sigma v^2}{n-1}}$$

as explained on page 14 of this paper. The error u, corresponding to a probability of one in one thousand, was also computed, the results being given in Table 3 following.

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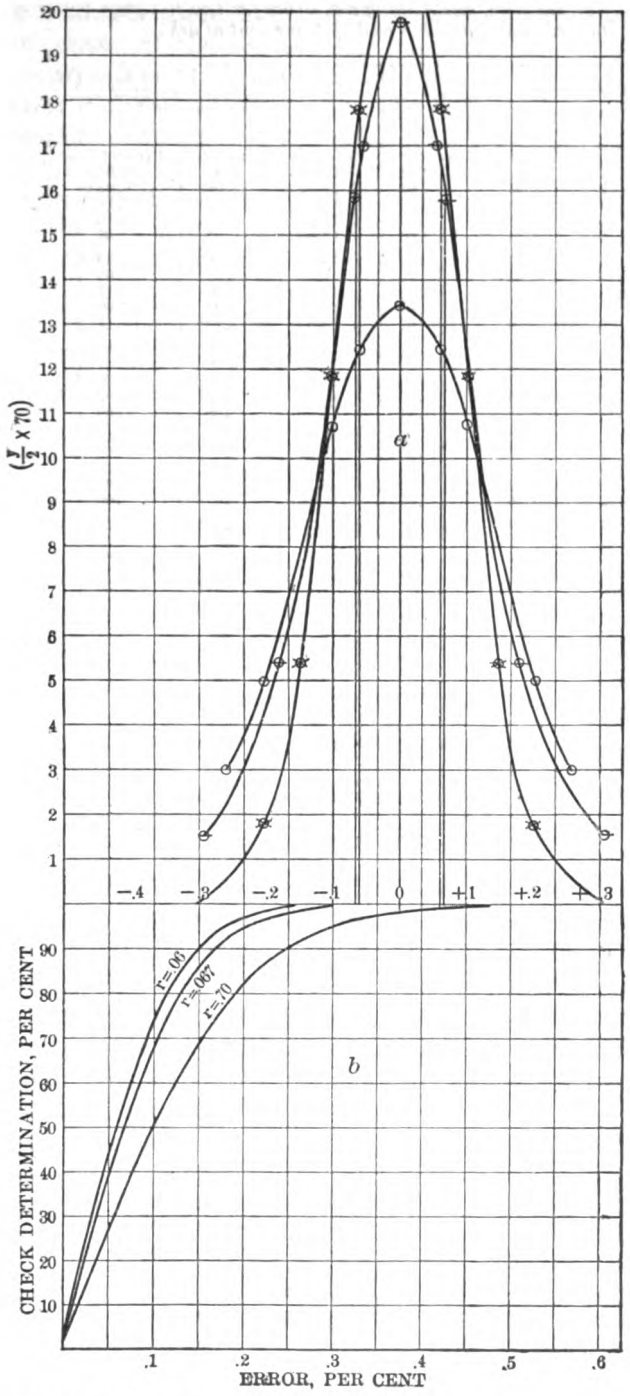

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FIGURE 3.—Ash curves.

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TABLE 3.—*Results of 24 analyses, establishing ash errors for two 100-gram bottle samples of coal containing 8.8 and 13.6 per cent of ash.*

Analysis No.	Ash.	Residual v.	Ash.	Residual v_1.	Residual v_2.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	8.92	0.05	13.70	0.03	0.04
2	8.80	.07	13.71	.04	.05
3	8.95	.08	13.55	.12	.11
4	8.85	.02	13.50	.17	.16
5	8.93	.06	13.60	.07	.06
6	8.80	.07	13.85	.18	.19
7	8.68	.19	13.75	.08	.09
8	8.85	.02	13.77	.10	.11
9	8.90	.03	13.58	.09	.08
10	8.80	.07	13.80	.13	.14
11	8.85	.02	13.82	.15	.16
12	8.85	.02	13.93	a.26	.....
13	8.87	.00	13.72	.05	.06
14	8.93	.06	13.64	.03	.02
15	8.98	.11	13.75	.08	.09
16	8.85	.02	13.77	.10	.11
17	8.87	.00	13.81	.04	.05
18	8.90	.03	13.65	.02	.01
19	8.93	.06	13.70	.03	.04
20	8.90	.03	13.60	.07	.06
21	8.90	.03	13.70	.03	.04
22	8.90	.03	13.57	.10	.09
23	8.95	.08	13.72	.05	.06
24	8.85	.02	13.77	.10	.11
Mean...	8.875	.....	.....	13.674	13.66

a Rejected by Chauvenet's criterion; the deviation from the r as first found is $r \times 3.43$, or 0.24 per cent.

$$r=0.6745\sqrt{\frac{\Sigma v^2}{n-1}}=0.043 \text{ per cent.}$$

$$u=4.9r=0.21 \text{ per cent.}$$

Using v^2

$$r=0.6745\sqrt{\frac{\Sigma v^2}{n-1}}=0.067 \text{ per cent.}$$

$$u=4.9r-0.33 \text{ per cent.}$$

The results in Table 3 mean that for series of determinations with coals of the same character and ash content as those used in the experiments, half of the errors for one might be expected to fall within the limit of 0.043 per cent, and for the other within the limit 0.067 per cent; once in one thousand determinations an error of 0.21 per cent might be expected for the one and of 0.33 per cent for the other. The order of accuracy obtainable is thus defined. The distribution of errors for the sample of Table 3 which contains 13.66 per cent ash is given in Table 4 (p. 19) with the distribution of errors of the series, 1,000 check samples taken from the records. The theoretical number of errors shown in Table 4 were calculated (r, the probable error being known) from the formula

$$P=\frac{2}{\sqrt{\pi}}\int_0^t e^{-t^2}dt$$

referred to on page 13 of this paper. The values of the integral as taken from the tables range from 0 to 1 and each value read for the

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x value corresponding may be taken as a fraction of the total number of errors of the series. For example, for the series for which the mean ash is 13.66 per cent, the value of h was calculated from equation 3 ($hr=0.4769$) whence the value of $hx=t$ of the integral was found. Substituting the value of 0.1 per cent for x (the value of $hx=t$ is 0.7 per cent) the corresponding probability is found from the tables to be 0.6778. Now there are 24 determinations (and hence 24 errors) in the series, and 24 times 0.6778 gives 16 as the theoretical number of errors for the series within the limit of 0.1 per cent. Another way of stating this result is that 67.78 per cent of the total number of errors of the series fall within 0.1 per cent. For the curves in *b*, fig. 3, the values for the ordinates were calculated for corresponding values of a by the method just given. Curves in x, fig. 3, were calculated from the formula

$$y = \pi^{-\frac{1}{2}} he^{-h^2x^2}$$

as explained on page 15 of this paper, each curve being designated by the value of r, the probable error of the series corresponding.

TABLE 4.—*Results establishing distribution of error in ash determinations.*

[$r=0.06$ per cent, computed from a table of values for the probability integral, $P=\frac{2}{\sqrt{\pi}}\int_0^t e^{-t^2}\,dt$ for argument $t=\frac{x}{r}$]

1000 CHECK SAMPLES WITH ASH UP TO 12.5 PER CENT.

Limits, per cent.	Errors found.	Theoretical number of errors.	Theoretical in 1000.
0.0 to −0.1	943	739	.....
.0 to − .2	979	975	.....
.0 to − .3	993	999	.....
.0 to − .4	996	1,000	.....
.0 to − .5	999	.....	.....

24 DETERMINATIONS ON 100-GRAM BOTTLE SAMPLE CONTAINING 13.66 PER CENT ASH.

0.00 to −0.05	6	8	385
.00 to − .10	14	16	685
.00 to − .15	20	19	869

The significance of these curves may be understood by considering that for a given value of x, the number of errors of the series falling within limits is proportional to the area included between the curve, the axes, and the ordinates for $+x$ and $-x$. As will be seen, the ordinate y measures the precision, or, rather, gives the relative precision of the series plotted to the same scale as represented by the curves. The steep curves will clearly represent the greater number of small errors, and will therefore indicate the greater precision. In curves *a*, figure 3, the curve representing the series $r=0.06$ per cent shows this series to be the most precise of the four represented, prac-

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tically all of the errors of the series being included by the curve and the ordinates corresponding to values +0.3 per cent and −0.3 per cent for the error x, and one-half of the errors within values +0.06 per cent and −0.06 per cent. The other two curves shown ($r=0.067$ per cent and 0.10 per cent) will be seen to represent a lower degree of precision. It will be noted that there are two series for which $r=0.10$ per cent and since this is so both are represented by the same curve.

As the foregoing explanation applies to all error-distribution curves given in this paper, the theory of such curves, presented subsequently, will be clear without further discussion.

TABLE 5.—*Results establishing error of riffling and of ash determination, 12 successive samples of 5-pound 20-mesh sample.*

Sample.	Ash.		Mean.	Moisture.	Mean.	Maximum.	Minimum.
	Determination A.	Determination B.					
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	16.55	16.35	16.45	2.70–2.70	2.70	2.73	2.45
2	16.45	16.55	16.48	2.70–2.75	2.73	.....	.....
3	16.55	[a] 16.95	16.75	2.75–2.60	2.68	.....	.....
4	16.80	16.55	16.68	2.70–2.65	2.68	.....	.....
5	16.85	16.60	16.73	2.80–2.70	2.75	.....	.....
6	16.50	16.55	16.53	2.65–2.70	2.68	.....	.....
7	16.50	16.40	16.45	2.60–2.55	2.58	.....	.....
8	16.75	16.70	16.73	2.70–2.55	2.63	.....	.....
9	16.45	16.40	16.43	2.55–2.65	2.60	.....	.....
10	16.55	16.65	16.60	2.65–2.55	2.60	.....	.....
11	16.50	16.45	16.48	2.55–2.50	2.53	.....	.....
12	16.80	16.95	16.88	2.50–2.40	2.45	.....	.....
Mean.	.....	.....	16.60	.....	.....	.....	.....

[a] Checked by third determination giving 16.75 per cent.

$$r=0.6745\sqrt{\frac{\Sigma v^2}{n-1}}=0.103 \text{ per cent.}$$

(from residuals, Mean = 16.60 − mean of determinations A and B for samples 1 to 12).

$$u=4.9r=0.50 \text{ per cent.}$$

Table 5 gives the results of 12 ash determinations of successive samples taken from the same 5-pound gross sample, the probable error found being 0.103 per cent. This value includes, of course, the error of the ash determination and that from sampling. The error applies only to the particular coal examined, and in order to answer the question as to what the figure would be for the average coal containing up to that amount of ash, the records of 1,000 consecutive check determinations were noted; each of the differences between the results for the first sample and those for the duplicate sample was halved; and the residuals thus obtained were squared and added. The value for r, found by the usual method, was 0.10

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per cent, or practically the same as that found for the results of 12 determinations given above.

It will be appreciated that the 1,000 check determinations do not represent 1,000 observations of the same quantity, but rather a second observation of each of 1,000 quantities differing slightly in value, the mean of each pair of observations being taken as the true value. As the errors of observations for all pairs of the series are of the same order of magnitude, the law of residuals

$$r = 0.6745\sqrt{\frac{\Sigma v^2}{n-1}}$$

will apply. In the application of the formula here it should, of course, be considered that for every pair of observations there are two residuals equal in value and opposite in sign. For example, if one observation gives a value of 14.20 per cent and the duplicate 14.40 per cent, the mean would be 14.30 per cent and the residuals would be +0.10 per cent and −0.10 per cent. Now if we call n of the formula the number of check determinations, and v half the difference between duplicates, we must write for the probable error

$$r = 0.6745\sqrt{\frac{2\Sigma v^2}{2n-1}}.$$

However, if n is large practically the same result is obtained from the formula

$$r = 0.6745\sqrt{\frac{\Sigma v^2}{n-1}},$$

which is accordingly used here.

It might be expected that the result for the 1,000 check samples would be lower than that for the individual sample, as the check samples included a large number of low-ash coals. However, this would be offset to some extent, as variation in moisture content was not considered in connection with the 1,000 check samples. The curve for this series of 1,000 check samples ($r = 0.10$ per cent) is shown in figure 3, page 17. It will be noted that the series for 12 determinations ($r = .10$ per cent) is represented by the same curve.

The ash in approximately 85 per cent of the coals analyzed in the laboratory does not exceed 12 per cent, and it was thought desirable to determine the probable error that would represent coals containing not to exceed 12 per cent ash. The figure was found to be 0.067 per cent calculated from the records of 1,000 check determinations, the method of computation being the same as that for the 1,000 check determinations referred to above. However, only determinations for the same bottle sample were considered in this calculation.

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Data for 106 sets of duplicate samples analyzed in the bureau's laboratory at Pittsburgh, Pa., are given in Table 6, following.[a]

The differences between duplicates given in this table were halved and the results squared. The sum of these squared residuals was found to be 151.2650, which may be taken as Σv^2 of the formula for the probable error r,

$$r = 0.6745\sqrt{\frac{\Sigma v^2}{n-1}}.$$

Substituting the value found and 105 for $n-1$ of the formula and solving, r is found to equal 0.083 per cent. The value of the error corresponding to a probability of one in one thousand (4.9 r) is found to be 0.406 per cent.

TABLE 6.—*Results establishing the ash error in the analyses of 106 sets of duplicate samples.*[b]

Laboratory No.	Kind of coal.	Source.	Ash in moisture-free coal.		
			Original.	Reserve part.	Difference.
			Per cent.	*Per cent.*	*Per cent.*
.....	Lignite.....	Valley County, Mont.....	.....	.....	.....
10900	.....do.....	.....do.....	6.99	7.30	0.31
11066	.....do.....	.....do.....	9.54	9.90	.36
10914	.....do.....	Dawson County, Mont.....	9.24	9.33	.09
11361	.....do.....	.....do.....	7.91	8.01	.10
11294	.....do.....	Wood County, N. Dak.....	11.15	10.94	.21
11048	Subbituminous.....	Converse County, Wyo.....	6.42	6.35	.07
11059	.....do.....	Johnson County, Wyo.....	18.02	18.32	.30
10827	.....do.....	.....do.....	6.75	7.18	.43
10671	.....do.....	Natrona County, Wyo.....	7.60	7.70	.10
10825	.....do.....	Sheridan County, Wyo.....	5.88	6.22	.34
12006	.....do.....	.....do.....	4.70	4.86	.16
10713	.....do.....	Uinta County, Utah.....	9.03	9.17	.14
10997	.....do.....	Wasatch County, Utah.....	3.57	3.70	.13
11058	.....do.....	.....do.....	7.68	7.70	.02
10732	.....do.....	El Paso County, Colo.....	20.77	21.33	.56
12099	.....do.....	.....do.....	8.75	8.71	.04
11114	.....do.....	Philippine Islands.....	8.17	8.69	.52
16134	Subbituminous slack...	Hot Springs County, Wyo.....	6.17	6.03	.14
16136	.....do.....	.....do.....	5.94	5.94	.....
16138	.....do.....	.....do.....	5.80	5.75	.05
16517	.....do.....	.....do.....	5.43	5.54	.11
16528	.....do.....	.....do.....	5.72	5.46	.26
16530	.....do.....	.....do.....	5.92	5.66	.26
16689	.....do.....	.....do.....	5.70	5.89	.19
12085	Bituminous.....	Jefferson County, Ala.....	10.91	11.38	.47
12196	.....do.....	Las Animas County, Colo.....	11.75	11.92	.17
11352	.....do.....	Montgomery County, Ill.....	11.47	11.45	.02
14111	.....do.....	Saline County, Ill.....	7.98	7.77	.21
16052	Bituminous, run of mine.	Vermillion County, Ill.....	12.02	12.29	.27
16053	.....do.....	.....do.....	11.83	11.95	.12
16054	.....do.....	.....do.....	12.07	12.04	.03
12004	Bituminous.....	Pike County, Ky.....	2.75	3.04	.29
11267	.....do.....	Henry County, Mo.....	16.80	16.78	.02
11268	.....do.....	.....do.....	14.26	14.23	.03
11269	.....do.....	.....do.....	11.66	11.96	.30
11499	.....do.....	Randolph County, Mo.....	9.28	9.19	.09
11918	.....do.....	Pittsburgh County, Okla.....	4.57	4.24	.33

[a] Fieldner, A. C., Notes on the sampling and analysis of coal: Tech. Paper 76, Bureau of Mines, 1914, pp. 53-55.

[b] The sample received at the laboratory, after having been crushed to pass a 10-mesh screen, is divided into two parts, one part being pulverized in the ball mill for the regular sample, and the other part being placed back in the can and held in reserve.

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TABLE 6.—*Results establishing the ash error in the analyses of 106 sets of duplicate samples*—Continued.

Laboratory No.	Kind of coal.	Source.	Ash in moisture-free coal.		
			Original.	Reserve part.	Difference.
			Per cent.	*Per cent.*	*Per cent.*
16481	Pittsburgh slack	Allegheny County, Pa	12.49	12.90	0.41
16681	do	do	15.57	15.41	.16
16483	do	do	11.94	12.02	.08
16693	do	do	11.96	12.54	.58
16485	do	do	12.50	12.80	.30
16912	do	do	11.95	12.05	.10
16049	do	do	10.24	10.40	.16
16050	do	do	10.29	10.43	.14
16051	do	do	9.80	9.80	.00
10845	Bituminous	do	6.13	5.99	.14
12572	do	do	5.92	5.97	.05
12105	do	Indiana County, Pa	9.21	9.30	.09
11875	Bony coal	do	21.31	21.23	.08
13930	Pittsburgh slack	Westmoreland County, Pa	11.24	11.12	.12
13932	do	do	11.13	11.25	.12
13934	do	do	10.99	10.86	.13
13936	do	do	10.93	10.98	.05
13944	do	do	11.04	11.04	.....
13946	do	do	11.00	11.00	.....
13948	do	do	11.08	11.01	.07
13950	do	do	11.23	11.16	.07
10801	Bituminous	Bledsoe County, Tenn	7.52	7.47	.05
11050	do	do	6.89	7.25	.36
10692	do	Rhea County, Tenn	12.03	11.98	.05
10909	do	Carbon County, Utah	7.37	7.04	.33
10736	do	Russell County, Va	5.93	5.81	.12
10739	do	do	5.44	4.86	.58
11736	do	King County, Wash	11.38	11.54	.16
10575	do	Pierce County, Wash	15.32	15.00	.32
16415	do	Weston County, Wyo	16.84	17.10	.26
16431	do	do	18.40	18.60	.20
14034	do	West Virginia	7.94	7.72	.22
14035	do	do	6.78	6.60	.18
11430	do	Marion County, W. Va	6.08	6.25	.17
14488	do	Marshall County, W. Va	7.26	7.16	.10
11744	do	Japan	5.03	4.75	.28
16700	do	do	5.63	5.69	.06
14038	do	do	13.37	13.01	.36
14040	do	do	13.20	13.30	.10
16936	do	China	15.92	16.39	.47
13270	do	Westmoreland County, Pa	9.58	9.47	.11
12183	Semibituminous	Cambria County, Pa	6.15	6.45	.30
10623	do	New River, Fayette County, W. Va.	2.83	2.56	.27
10628	do	New River, Raleigh County, W. Va.	3.65	3.58	.07
10630	do	do	3.70	3.64	.06
12237	do	Pocahontas bed, McDowell County, W. Va.	11.32	11.38	.06
12036	Anthracite	Pennsylvania	12.22	12.03	.19
17053	do	do	25.45	25.03	.42
11782	do	Luzerne County, Pa	7.23	7.05	.18
11441	do	do	6.03	5.89	.14
11440	do	Lackawanna County, Pa	11.94	11.66	.28
13952	Anthracite, No. 1 buckwheat.	Pennsylvania	17.55	17.45	.10
13954	do	do	17.48	17.11	.37
13956	do	do	17.03	17.27	.24
13958	do	do	17.02	17.21	.19
13963	do	do	17.60	17.35	.25
13967	do	do	17.77	17.53	.24
13965	do	do	17.38	18.11	.73
13969	do	do	17.30	17.33	.03
13905	Anthracite, No. 3 buckwheat.	do	18.65	18.37	.28
13907	do	do	18.67	18.69	.02
13909	do	do	19.13	19.00	.13
13911	do	do	18.96	18.84	.12
13915	do	do	18.11	18.43	.32
13917	do	do	18.42	18.56	.14
13919	do	do	17.49	17.49	.00
13921	do	do	17.71	17.68	.03
13926	do	do	17.62	17.60	.02
13928	do	do	17.91	17.96	.05

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This series, as will be seen, showed slightly higher precision than was shown by the series of 1,000 check determinations including coals containing up to 17 per cent ash; however, in the latter the moisture variation was not considered and consequently the result might be expected to be somewhat high.

Now in this series, for which r equals 0.083 per cent, there were five pairs of duplicates where the variation was as much as 0.5 per cent. It will be interesting to calculate for the series the number required by theory. A variation of 0.5 per cent between duplicates means that for each determination the variation from the mean is 0.25 per cent; therefore we may consider that we have 10 determinations where the error is 0.25 per cent. Knowing the value of r (0.083 per cent) for the series we may read from the tables of the probability integral $\frac{2}{\sqrt{\pi}}\int_0^t e^{-t^2}dt$, any value of x of the series. In this instance, where x equals 0.25 per cent, the probability is 0.9577, which means that 95.77 per cent of the errors of the series are within limit 0.25 per cent. There are 106 duplicate determinations for the series, hence 212 single determinations and 212 errors. As 95.77 per cent of 212 equals 203, 203 errors less than 0.25 per cent are required for the series by theory; that is, 101.5 pairs of duplicates. Then $106-101.5=4.5$ which should vary more than 0.5 per cent, required by theory, whereas 5 were actually found. Clearly the agreement is close. A wider difference between theory and practice might have been expected because the average mean used must have differed somewhat from the true mean and because the true ash can not be accurately defined.

It would seem almost axiomatic that determinations for coals of high ash content would be subject to greater error than those for coals of low ash content, although it is easily seen that the ash distribution, and therefore the difficulty with some coals of obtaining a sample representative of the impurities present, might alter this conclusion. That the proposition is true in general, however, is indicated by the following series:

(1) For ash under 5 per cent, a series of 42 check determinations gives r equals 0.027 per cent.

(2) For ash from 5 per cent to 10 per cent, a series of 103 check determinations gives r equals 0.033 per cent.

(3) For ash from 10 per cent to 15 per cent, a series of 66 check determinations gives r equals 0.124 per cent.

(4) For a series of 67 check samples in which the ash was 15 to 25 per cent the probable error was 0.20 per cent.

Series 4 includes the error of the ash determination and that of sampling, and may be taken to indicate about what may be expected for check samples of coals containing the maximum amount of ash.

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ERROR IN MOISTURE DETERMINATION.

The method followed in the Bureau of Mines laboratory in making a moisture determination is as follows:

One gram (±0.2 milligram) of the sample is weighed into a No. 2 porcelain capsule which is provided with a numbered thin aluminum lid. The capsule, uncovered, is placed in a glycerin-jacketed oven and dried at a temperature of 105° to 110° C. for 1 hour. During the drying, warm air, previously dried by bubbling through concentrated sulphuric acid, is passed through the drying compartment of the oven at the rate of about 4 volumes a minute. The capsule containing the dried coal is then taken from the oven, covered, cooled in a desiccator, and weighed. The first weighing of the sample, as well as the transfer and weighing of the dried sample, is effected as quickly as possible in order to prevent the sample from taking up moisture through exposure. In this connection the authors wish to state that they have found it comparatively easy to weigh to 0.2 milligram without introducing an error of greater magnitude through changes in moisture content of the sample.

The weight of the sample as well as subsequent weights may be quickly obtained by "snap weighing" or weighing to a standstill—that is, adjusting the weights till the balance pointer remains at zero without swinging.

Reasonable accuracy in "snap weighing" requires considerable practice; also a quick accurate balance is absolutely essential; its sensibility should be high. Most analytical balances are not satisfactory for "snap weighing"; they are too slow and do not permit the required delicate adjustment. A light balance that will weigh 50 to 100 grams and is sensitive to one-twentieth milligram is to be preferred and it must be so adjusted that it will quickly indicate an excess of 0.1 milligram from standstill. It is well, of course, to practice making "snap weighings" with some substance not subject to moisture changes, as the weight first obtained may then be checked.

Table 7 following shows the results of two series of determinations for moisture—one series for a coal having a moisture content of 1.03 per cent and one for a coal containing 8.50 per cent moisture.

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TABLE 7.—*Results establishing the moisture error for two 100-gram bottle samples containing 1.03 and 8.50 per cent moisture.*

Test No.	Series 1.		Series 2.	
	Moisture.	Residual v.	Moisture.	Residual v.
1	0.98	−0.05	8.55	0.05
2	1.02	− .01	8.59	.09
3	.92	− .11	8.45	− .05
4	.98	− .05	8.50	.00
5	.95	.08	8.45	− .06
6	1.00	− .03	8.55	.05
7	1.03	.00	8.60	.10
8	.98	− .05	8.61	.11
9	1.06	.03	8.65	.15
10	1.15	.12	8.60	.10
11	1.10	.07	8.43	− .07
12	1.06	.03	8.40	− .10
13	1.10	.07	8.48	− .02
14	1.05	.02	8.45	− .05
15	1.05	.02	8.33	− .17
16	1.05	.02	8.35	− .15
17	1.05	.02	8.60	.10
18	1.05	.02	8.58	.08
19	1.07	.04	8.60	.10
20	.98	− .05	8.45	− .05
21	1.02	− .01	8.38	− .12
22	1.07	.04	8.45	− .05
23	1.05	.02	8.40	− .10
Mean	1.034		8.502	

$r=0.6745\sqrt{\frac{\Sigma v^2}{n-1}}=0.034$ per cent. $r=0.6745\sqrt{\frac{\Sigma v^2}{n-1}}=0.064$ per cent.

$u=4.9r=0.17$ per cent. $u=4.9r=0.31$ per cent.

The distribution of errors for the series of tests covering the sample with a moisture content of 8.502 per cent was computed in the same manner as the distribution of errors shown in Table 4 (p. 19), being based on the following values:

Results establishing distribution of error in analyses of 100-gram bottle sample of coal containing 8.502 per cent moisture.

Limits, per cent.	Errors found.	Theoretical number.	Theoretical number in 1,000.
0.00 to −0.05	8	8	385
.00 to − .10	19	17	685
.05 to − .15	23	19	869

All the determinations were made from the same 100-gram bottle sample as was used in the ash determination represented in Table 3 (p. 18), and there seems to be nothing deserving of particular notice except that there is no preponderance of negative residuals with the last determinations of either series. It might be expected that the last samples weighed would run low in moisture, as it was necessary to expose the whole sample repeatedly while the first samples were

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being weighed; and it may be stated further that none of the samples had been previously air dried. It would seem, then, that there is in reality not much moisture change in the gross sample during the weighing of the moisture sample.

It is the practice of the Bureau of Mines laboratory to make a separate moisture determination from a 20-mesh grab sample taken immediately after the 5-pound sample has been crushed to 20 mesh, a 5-gram sample being weighed out for the determination. No figures were obtained for the possible precision with this 5-gram 20-mesh sample, but it was noted in a general way, while the data on 400 check determinations for the 60-mesh sample were being taken from the records, that the agreement for the determinations with the 20-mesh sample was better than that for the 60-mesh sample. As the 20-mesh sample almost invariably contains the most moisture, some of which may be extraneous,[a] it would seem that, in general, errors of the moisture determination are made up mostly of errors in weighing and are not largely due to gain or loss through exposure. The weighing error connected with the 5-gram 20-mesh sample is, of course, divided by 5, whereas an exposure error might be expected to vary as the area exposed.

If the size of the particles can be said to have an influence on the tendency of a coal to retain or lose moisture during exposure, the 20-mesh sample might be expected to change more rapidly than the 60-mesh sample. As in reality the 20-mesh samples considered here consist mostly of finer coal (Table 1, p. 9) the effect of the coarser particles would be to keep the sample from packing readily and thus to leave more of the fine particles, which are conceived to retain the most extraneous moisture, exposed to air circulation.

The probable error for the 60-mesh coal in 400 check determinations represented in the records was 0.034 per cent, 95 per cent of the determinations showing a moisture content of less than 4 per cent. This degree of precision, as will be seen, is about the same as that obtainable in the ash determination with coals containing 5 to 10 per cent ash. Seemingly the error for the moisture determination increases more rapidly with increasing moisture content than does the ash error for a corresponding increase in ash content.

ERROR IN VOLATILE-MATTER DETERMINATION.

The method used by the authors for the determination of volatile matter was as follows:

One gram of the 60-mesh coal was weighed into a 15-c. c. platinum crucible provided with a closely fitting cover and heated for seven minutes in a small electrically heated muffle furnace kept at a tem-

[a] Fieldner, A. C., Notes on the sampling and analysis of coal: Tech. Paper 76, Bureau of Mines, 1914, p. 23.

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perature of 950° C. (±15° C.) by means of an automatic temperaure regulator. The crucible containing the residue was then cooled on a heavy cast-iron plate and weighed. The loss in weight minus the moisture found for the sample was taken as the volatile matter. The crucibles were always placed in the same relative position in the furnace, being placed as near as possible to the regulating thermocouple. However, the temperature in a considerable part of the furnace does not vary appreciably from that at the point of the regulating couple, as the authors have demonstrated by exploring the furnace with a quick-acting auxiliary pyrometer.

The results of a series of 24 determinations on a 100-gram 60-mesh sample of semibutuminous coal are given in Table 8 following:

TABLE 8.—*Results establishing volatile-matter error for 100-gram bottle sample of semibituminous coal having a volatile-matter content of 19.32 per cent.*

Test No.	Volatile matter, per cent.	Residual v.	Test No.	Volatile matter, per cent.	Residual v.
1	19.75	0.43	14	19.80	0.52
2	19.50	.18	15	19.30	−.02
3	19.00	−.32	16	19.15	−.17
4	19.60	.28	17	19.55	.23
5	19.70	.38	18	19.10	−.22
6	18.95	−.37	19	19.75	.43
7	19.30	−.02	20	19.25	−.07
8	19.25	−.07	21	19.25	−.07
9	19.65	.33	22	19.10	−.02
10	19.45	.13	23	19.30	−.02
11	19.50	.18	24	19.20	−.12
12	19.30	−.02	Mean	19.32	
13	19.15	−.17			

$$r=0.6745\sqrt{\frac{\Sigma v^2}{n-1}}=0.18 \text{ per cent.}$$

$$u=4.9r=0.86 \text{ per cent.}$$

Distribution of errors.

Limit, per cent.	Errors found.	Theoretical number.	Theoretical number in 1,000.
0.10 to −0.10	6	7	300
.20 to − .20	13	13	559
.30 to − .30	17	18	752
.40 to − .40	21	21	876
.50 to − .50	23	24	946

Curves based on the results presented in Table 8 are shown in figure 4. A series of 500 check determinations contained in the laboratory records gave a probable error of 0.104 per cent. That the error for the large series is less than that for the 24 determinations on the same sample is perhaps due to the fact that the bituminous coal used in the smaller series gave off its volatile more rapidly than does the average coal, causing greater variations. In this connection it may be stated that for the series of 500 check

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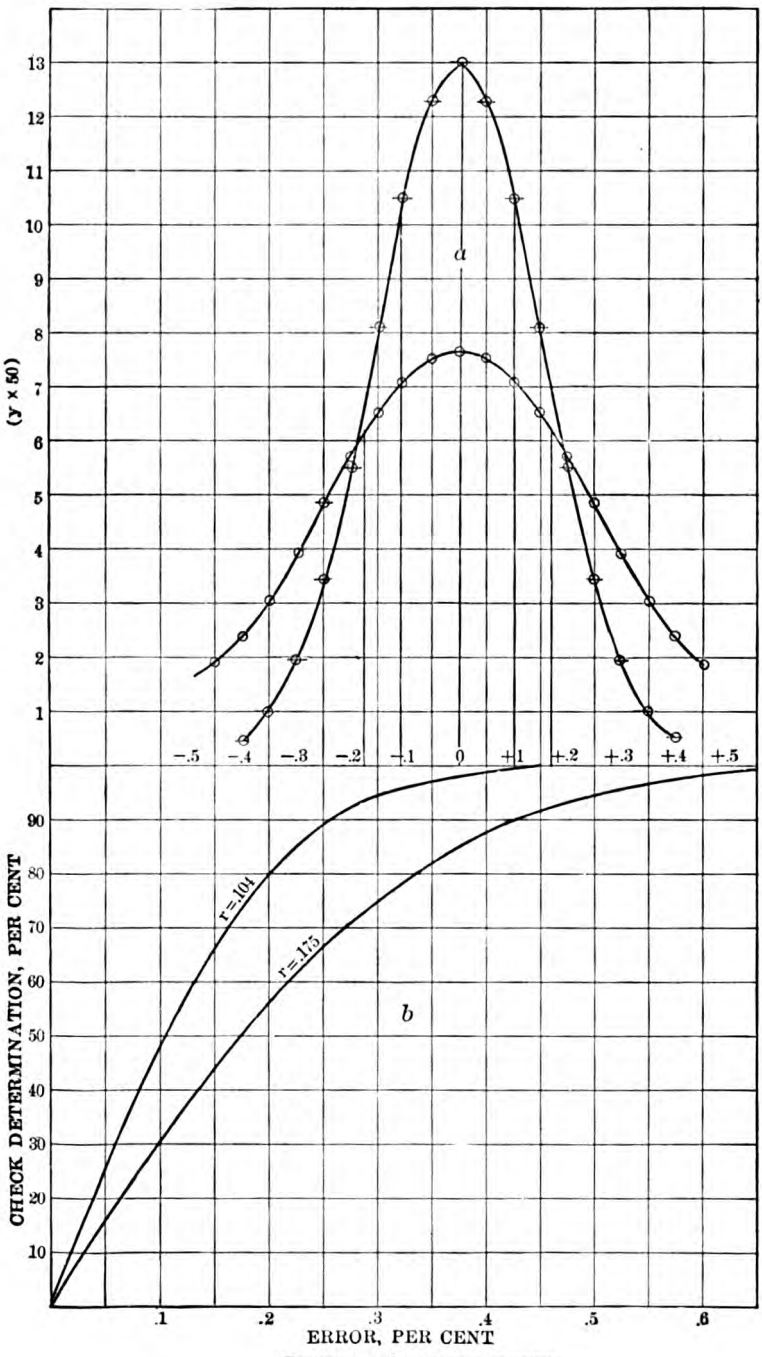

FIGURE 4.—Volatile matter curves.

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determinations no determinations originally rejected were considered. The determinations rejected represented tests in which explosions occurred or in which other errors were evident. The effect of an explosion in the crucible is to lift the lid of the crucible, thereby admitting air which oxidizes somewhat the coke residue, making the result for the determination too high.

In the experience of the authors the use of the electric furnace is attended with more trouble from "explosions" than is the use of the Bunsen burner. With the furnace the temperature within the crucible comes to a maximum more quickly, and any large amount of volatile matter present is evolved with explosive force, whereas with the burner the evolution of volatile matter takes place more slowly. All things considered, however, the furnace gives the best results as it is capable of closer temperature regulation. For constancy of results it has been shown [a] that control of the temperature of the distillation within narrow limits is essential, and although a method might be evolved that would obviate the explosion trouble for a particular kind of coal, such a method would probably be subject to more serious error in other respects.

ERROR IN HEATING-VALUE DETERMINATION.

In the Bureau of Mines laboratory the heating-value determination is made with an acid-proof oxygen bomb and a water-jacketed calorimeter designed especially for the bureau's work. The precision attainable is somewhat higher than that obtained with the usual form of fuel calorimeter; further, the time required for manipulation and observation is considerably shorter than with many other types.

Owing to the seeming simplicity of the heating-value determination, the results obtained in the determination may be assumed to be of unquestioned accuracy when such an assumption is not warranted. The determination requires fully as much manipulative skill and close attention to details as does the carbon-hydrogen determination, and the effect of the personal equation is therefore apt to be large in comparison with the effect of errors attributable to the instrument used.

The results given in Table 9 following are regarded as indicating the best work possible with the bureau's instrument in the hands of a particularly skillful manipulator.

[a] Fieldner, A. C., and Hall, A. E., Influence of temperature on the determination of volatile matter in coal: Proc. Eighth Internat. Cong. App. Chem., vol. 10, 1912, p. 139.

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TABLE 9.—*Results establishing the heating-value error in 24 determinations on bottle sample of 100 grams of coal.*

Date of determination.	Heating value.	Residual v.	v^2.
1914:	*B. t. u.*	*B. t. u.*	
Jan. 22	14,651	5	25
24	14,639	−7	49
26	14,644	−2	4
27	14,653	7	49
28	14,648	2	4
29	14,649	3	9
30	14,646	0	0
Feb. 2	14,649	3	9
3	14,649	3	9
4	14,639	−7	49
5	14,644	−2	4
6	14,648	2	4
7	14,639	−7	49
9	14,641	−5	25
11	14,653	−7	49
12	14,651	−5	25
13	14,644	−2	5
14	14,653	7	49
16	14,644	−2	4
17	14,644	−2	4
18	14,651	5	25
19	14,644	−2	4
20	14,639	−7	49
24	14,639	−7	49
Mean	14,646		

$$\Sigma v^2 = 551$$

$$r = 0.6745\sqrt{\frac{\Sigma v^2}{n-1}} = 3.2 \text{ B. t. u.}$$

$$u = 4.9r = 15.7 \text{ B. t. u.}$$

The curve representing the results presented in the table is given in figure 5. There are also plotted in figure 4 the curve for 1,000 check determinations where $r = 16$ B. t. u. and the curve for 500 check determinations where $r = 24.7$ B. t. u. The curve $r = 16$ B. t. u. includes variations due to the personal factor, and to differences in calibration of the different instruments, and indicates the precision to be expected under the conditions of regular routine work. The curve for $r = 24.7$ B. t. u. includes all errors considered in the series $r = 16$ and in addition the widest variations occurring in the series, no determinations being rejected; that is, a number of observations known to have large constant errors were included. This series indicates merely what would happen if no rule for the rejection of doubtful observations were available and no attention were paid to such sources of error as slight amounts of unburned coal, thermostat setting slightly in error, excessive contact resistance in the thermometer switch, or a mistake of 0.001 ohm in the bridge reading.

As the determined heating value is a function of the moisture and ash content of the sample, it is of interest to calculate the heating value of the coal substance and to determine the effect of the combined errors for ash, moisture, and heating value on the calculated result.

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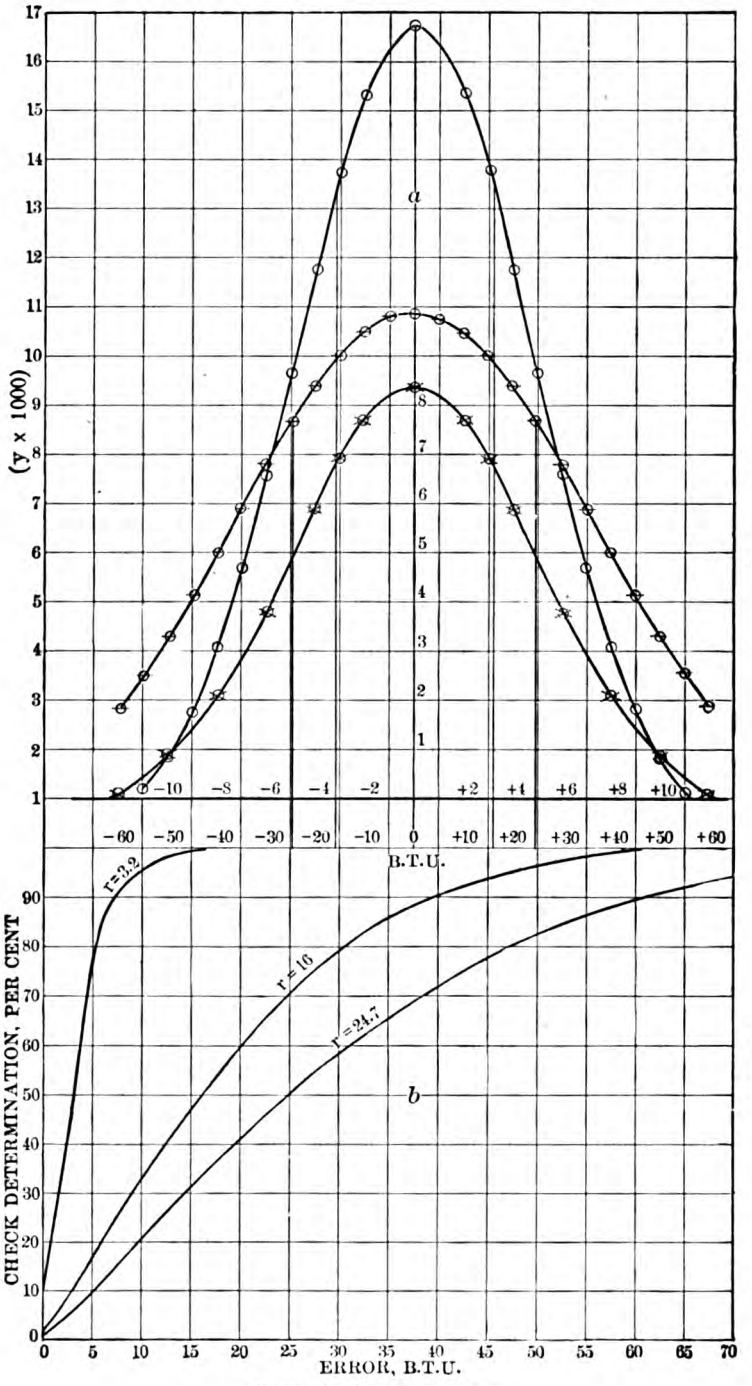
(y × 1000)
a
b
B.T.U.
−60 −50 −40 −30 −20 −10 0 +10 +20 +30 +40 +50 +60
−10 −8 −6 −4 −2 +2 +4 +6 +8 +10
CHECK DETERMINATION, PER CENT
ERROR, B.T.U.
r=9.2
r=16
r=24.7
For group a:
y=ke^{-h²t²}
r=16 B. t. u.; h=0.0298; 1,000 check analyses.
r=21.7 B. t. u.; h=0.0193; 500 check analyses.
r=3.2 B. t. u.; h=0.149; 24 check analyses, same sample.
For group b:
y=2π^{-½} ∫₀^t e^{-t²} dt
t=hr.

FIGURE 5.—Heating value curves.

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If variations from the true ash and moisture values be neglected the heating value of the coal substance is given by the formula:

$$B=\frac{A}{100-(x+y)} \tag{1}$$

Where B=calculated heating value, on ash-free and moisture-free basis
A=heating value as determined.
x=value found for ash.
y=value found for moisture.

The following assumption may be made:

r_1=0.15 per cent for the probable error in A.
r_2=0.10 per cent for the probable error in x (including sampling error).
r_3=0.04 per cent for the probable error in y (including sampling error).
R=the combined errors.

The combined error R, corresponding to the error in B, is given by the following formula, [a]

$$R^2=r_1^2\left[\frac{\delta B}{\delta A}\right]^2+r_2^2\left[\frac{\delta B}{\delta X}\right]^2+r_3^2\left[\frac{\delta B}{\delta Y}\right]^2 \tag{2}$$

applied to the formula given above.

Differentiating:

$$R^2=r_1^2\left[\frac{1}{100-x-y}\right]^2+r_2^2\left[\frac{A}{(100-x-y)^2}\right]^2+r_3^2\left[\frac{A}{(100-x-y)^2}\right]^2 \tag{3}$$

Now, assuming $A=14{,}000$, $x=8.00$ per cent, and $y=2.00$ per cent, and substituting these values and the values found for r_1, r_2, and r_3 in equation 3, we have $R=\sqrt{0.00000272+0.00477+0.0298}=0.18$ (nearly), which is the combined error for a coal of the character assumed. Of course the result would vary for different coals, but the foregoing calculation shows that the error introduced by the combination is of the same order as that of the individual errors and is not at all serious. Variations found, then, for the calculated heating value of the coal substance must be due largely to variations in percentages of volatile inorganic constituents (moisture, CO_2 and SO_2) of the ash,[b] variations in composition of the coal substance itself, and, perhaps also to some degree, a variable affinity for moisture.

It has been shown [c] that the heating value of coal substance as calculated from analyses of coal samples from the same seam is subject to variations usually not greater than 1 per cent.[b]

CONCLUSION.

The results obtained through the application of the theory of probability would seem to indicate rather clearly what degree of precision may be expected with the coal analysis when the quantities determined

[a] Bartlett, D. P., The method of least squares, 3rd ed., 1915, p. 57; Merriman, Mansfield, A textbook on the method of least squares, 1901, p. 79.
[b] Parr, S. W., Chemical composition of Illinois coals: Illinois State Geol. Survey Bull. 16, 1910, p. 232.
[c] Fieldner, A. C., Notes on the sampling and analysis of coal: Tech. Paper 76, Bureau of Mines, 1914, p. 47.

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are defined by the actual methods used for the analysis. When the error corresponding to a probability of one-half is taken, the errors involved in sampling and analyzing coal may be taken to be as follows: In taking the 5-pound gross sample, about 0.04 per cent of sample, because of failure to include or exclude impurities represented, is ash in the analysis; in the ash determination, 0.08 per cent; in the moisture determination, 0.04 per cent; in the volatile-matter determination, 0.15 per cent, and in the heating-value determination, 0.15 per cent. Combining these possible errors makes a possible error in calculating the heating value of coal substance of about 0.18 per cent of the heating value. Now the limit determining rejection is often taken as the error whose probability is 1 in 1000, which is 4.9 times (or roughly 5 times) the probable error of a single determination; the outside limit for error in the above determinations may be easily calculated. The limits of error would then be as follows: For sampling, 0.20 per cent; for the ash determination, 0.40 per cent; for the moisture determination, 0.20 per cent; and for the heating-value determination, 0.75 per cent. It would seem, then, that the limits allowed by the committee on coal analysis of the American Society for Testing Materials (see p. 6) are not limits outside which, with ordinary care, determinations would never fall; it would seem rather that the committee means to designate limits within which a large percentage of the errors will fall.

ACKNOWLEDGMENT.

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